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IS 15072 (2002): Sodium Alpha Olefin Sulphonate for Cosmetic, Detergent and other Industrial Use - [CHD 25: Soaps and other Surface Active Agents]

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प्रसाधन सामग्री, अपमार्जक द्रव्य और अन्य औद्योगिक प्रयोजन के लिये सोडियम अल्फा ओलिफिन सल्फोनेट — विशिष्टि

Indian Standard

SODIUM ALPHA OLEFIN SULPHONATE FOR COSMETIC, DETERGENT AND OTHER INDUSTRIAL USE — SPECIFICATION

ICS 71.080.99; 71.100.40;71.100.70

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 2002 **Price Group 7**

Soaps and Other Surface Active Agents Sectional Committee, CHD 25

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Soaps and Other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

Sodium alpha olefin sulphonate (AOS) is an intermediate detergent raw material being produced and marketed in substantial quantity in India for the manufacture of synthetic detergent powders, tablets, bathing bars, combination laundry soap bar, liquid detergents and cosmetic products. It has also various industrial applications like emulsion polymerization, oil drilling operation, etc. The chemical is very rich in surface active properties and is used either alone or in combination with other anionic or non-ionic surface active agents.

Sodium alpha olefin sulphonate is manufactured by the reaction of sulphur trioxide with alpha olefin, followed by neutralization with caustic soda and high temperature hydrolysis of the neutralized product. The reaction of alpha olefin with sulphur trioxide produces a mixture of sulphonic acid and internal sultones. High temperature hydrolysis in alkaline medium converts these internal sultones into alkene sulphonate and hydroxy alkane sulphonate. Both these components have almost same properties. The material also contains some quantity of disulphonate.

Improper manufacturing conditions for sulphonation, neutralization and hydrolysis, may lead to the formation of high contents of sultones. Normally, delta sultone and gamma sultone can be present in the product. Delta sultone is known to be not harmful. But gamma sultone is a skin sensitizer and, therefore, the content of gamma sultone has been limited to 1 ppm, at which level, it is not harmful.

Sodium hypochlorite should not be used to bleach the AOS or the products incorporating AOS, due to risk of forming unsaturated sultones and chlorosultones which are known to be skin sensitizers.

Substantial assistance has been drawn from the following document while formulating this standard:

> Determination of 1-alkene-1,3 sultone in anionic surfactants by gas chromatography — Mass spectrometry — Shigcaki Matsutani

The composition of the Committee responsible for the formulation of this standard is given in Annex H.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 2 SEPTEMBER 2008
TO
IS 15072 : 2002 SODIUM ALPHA OLEFIN SULPHONATE FOR COSMETIC, DETERGENT AND OTHER INDUSTRIAL USE — SPECIFICATION

(*Page* 1, *clause* **5.2**) — Insert the following at the end:

'**5.2.1** *BIS Certification Mark*

The packages may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act,* 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.'

(CHD 25)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 OCTOBER 2007
TO
IS 15072 : 2002 SODIUM ALPHA OLEFIN SULPHONATE FOR COSMETIC, DETERGENT AND OTHER INDUSTRIAL USE — SPECIFICATION

(*Foreword, para* 4, *last sentence*) — Substitute the following for the existing:

'But gamma sultone is a skin sensitizer and, therefore, the content of gamma sultone has been limited to 70 ppb.'

[*Page* 2, *Table 1*, *SI No.* (viii), *col* 2] — Substitute 'ppb' *for* 'ppm'.

(CHD 25)

Reprography Unit, BIS. New Delhi, India

Indian Standard

SODIUM ALPHA OLEFIN SULPHONATE FOR COSMETIC, DETERGENT AND OTHER INDUSTRIAL USE — SPECIFICATION

1 SCOPE

This standard prescribes requirements and methods of sampling and test for sodium alpha olefin sulphonate for cosmetic, detergent and other industrial use.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
286 : 1978	Methods of sampling and test for soaps (*second revision*)
321 : 1964	Specification for absolute alcohol (*revised*)
1070 : 1992	Reagent grade water (*third revision*)
2263 : 1979	Methods of preparation of indicator solutions (*first revision*)
4956 : 1977	Specification for synthetic detergents for industrial purposes (*first revision*)
11601 (Part2) : 1992	Method of safety evaluation of synthetic detergent: Part 2 Method of test for skin sensitization potential of synthetic detergents (Guinea pig maximization test)
12795 : 1989	Specification for linear alkyl benzene
13933 : 1995	Method of test for ready biodegradability of surface active agents (Modified sturm test)

3 TYPES

The material shall be of the following three types depending on its physical form and chemical composition:

Type 1 — Powder or flakes,

Type 2 — Paste, and

Type 3 — Liquid.

4 REQUIREMENTS

4.1 Description

The material shall be in the form of free flowing powder/flakes, paste or clear liquid, free from visible dirt and impurities. It shall not give any unpleasant odour.

4.2 The material shall pass the test for skin sensitization potential when evaluated as per the method prescribed in IS 11601 (Part 2).

4.3 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be suitably packed in airtight containers as agreed to between the purchaser and the supplier.

5.2 Marking

The containers shall be legibly and indelibly marked with the following information:

a) Name and type of the material;
b) Indication of source of manufacture;
c) Net mass of the material;
d) Batch No. or Lot No. in code or otherwise;
e) Month and year of manufacture; and
f) (*CAUTION*:'DO NOT USE CHLORINE BLEACH ALONG WITH THIS PRODUCT'.

6 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

7 SAMPLING

7.1 General

The general precautions, scale of sampling and preparation of test samples, shall be as prescribed in **3.1**, **3.2** and **3.3** of IS 286 as applicable for liquid soap respectively.

7.2 Number of Tests

7.2.1 Tests for the determination of characteristics given at SI No. (i) and (ii) in Table 1 shall be conducted on each of the individual samples separately.

7.2.2 Tests for determination of all the remaining characteristics shall be conducted on the composite sample.

7.3 Criteria for Conformity

7.3.1 *For Individual Sample*

For each of the characteristics which has been determined on the individual samples (**7.2.1**) the mean ($\bar{X}$) and the range (R) of the test results shall be calculated as follows:

$$\textbf{Mean } \bar{X} = \frac{\textbf{The sum of test results}}{\textbf{Number of test results}}$$

Range (R) = the difference between the maximum and the minimum value of the test results.

7.3.1.1 The lot shall be deemed as conforming to the requirement if the expression ($\bar{X}$ - 0.6 R) is greater than or equal to minimum value given in Table 1, and($\bar{X}$ + 0.6 R) is less than or equal to maximum value given in Table 1.

7.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of other characteristics determined on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirement.

Table 1 Requirements for Sodium Alpha Olefin Sulphonate

(*Clauses*4.3, 7.2.1 and 7.3.1.1)

SI No.	Characteristic	Requirements			Method of Test, Ref to		
		type 1	Type 2	Type 3	Annex of IS 4956	Annex of this Standard	IS No.
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Active ingredient, percent by mass, *Min*	90	64	37	C & D	—	—
ii)	Moisture content. *Max*	3.0	—	—	B	—	—
iii)	Free alpha olefin, percent by mass, *Max*	3.0	3.0	1.5	—	A	—
iv)	Free alkali (as NaOH), percent by mass, *Max*	0.5	0.5	0.2	—	B	—
v)	Colour						
	a) of 5 percent active solution in Klett colorimeter, *Max*	100	100	100	—	C	—
	b) of 5 percent active solution in Lovibond 5 ¼" cell, Y+5R, *Max*	12	12	12	—	C	—
vi)	Total inorganic salts, percent by mass, *Max*	6.0	3.0	3.0	—	D	—
vii)	Disulphonate, on 100 percent active basis, ppm, *Max*	6.0	6.0	6.0	—	E	—
viii)	Gamma sultone on 100 percent active basis, ppm, *Max*	70	70	70	—	F	—
ix)	Total sultone on 100 percent active basis, ppm, *Max*	200	200	200	—	F	—
x)	Internal sultone on 100 percent active basis, ppm, *Max*	50	50	50	—	F	—
xi)	Biodegradability	To pass the test	To pass the test	To pass the test	—	—	13933

ANNEX A

[*Table* 1,*Sl No.* (iii)]

DETERMINATION OF FREE ALPHA OLEFIN

A-l REAGENTS

A-l.l Absolute Alcohol — *See* IS 321.

A-1.2 Dilute Ethyl Alcohol — 50 percent.

A-1.3 Petroleum Ether — 40 to 60°C boiling range.

A-2 PROCEDURE

A-2.1 Weigh accurately about 5 g of sample (sodium alpha olefin sulphonate) into a 250-ml beaker. Dissolve in 50 ml of alcohol. Add 50 ml of water. Transfer it into a 250 ml separating funnel. Extract four times with 50 ml of petroleum ether. Wash the extract with 50 ml of 50 percent alcohol.

A-2.2 Filter the ether layer through anhydrous sodium sulphate taken on Whatman filter paper No. 1 or equivalent, in a previously weighed 150-ml beaker. Boil off the ether on a water bath to about 20 ml. Keep the beaker on a warm hot plate (60 to 80°C) and evaporate petroleum ether with a gentle stream of nitrogen or air. Stop when the bottom of the beaker starts becoming warm. Wipe any condensed moisture from outside the beaker. Complete evaporation by blowing a gentle stream of nitrogen or air through the beaker at room temperature for 10 min precisely. Weigh the beaker immediately to determine the mass of the residue.

A-3 CALCULATION

$$\text{Free alpha olefin, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = massing of residue, and
M_2 = mass in g of the sample taken for the test.

ANNEX B

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF FREE ALKALI

B-1 REAGENTS

B-l.l Phenolphthalein Indicator — Dissolve 1 gin 100 ml of 95 percent rectified spirit.

B-1.2 Distilled Water — *See* IS 1070.

B-l.3 Standard Sulphuric Acid or Hydrochloric Acid — Approximately 0.1 N.

B-2 PROCEDURE

Weigh accurately about 4 to 5 g of the sample and dissolve in 100 ml of distilled water. Heat, if necessary. Add about 0.5 ml of phenolphthalein indicator solution and titrate with standard sulphuric acid or hydrochloric acid.

B-3 CALCULATION

$$\text{Free alkali (as NaOH), percent by mass} = \frac{4VN}{M}$$

V = volume in ml of standard sulphuric acid or hydrochloric acid used;
N = normality of standard sulphuric or hydrochloric acid; and
M = mass in g of the material taken for the test.

ANNEX C

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF COLOUR

C-l GENERAL

Colour can be determined by the following two methods:

a) Klett colorimeter (Method A), and
b) Lovibond tintometer (Method B).

C-2 KLETT COLORIMETER (METHOD A)

C-2.1 Apparatus — Klett colorimeter.

C-2.2 Procedure

Dilute the alpha olefin sulphonate with distilled water to give a solution containing 5 percent active matter. Fill the solution in 4-cm cell and using No. 42 filter, match the colour of the solution with the standard colour slides. Report as the Klett colour the number of matched slides.

C-3 LOVIBOND TINTOMETER (METHOD B)

C-3.1 General

This method determines the colour of the solution of sodium alpha olefin sulphonate by comparison with Lovibond glasses of known colour characteristics. The colour is expressed as the sum of the yellow and five times red slides used to match the colour in a 5 ¼ inch cell.

C-3.2 Apparatus

C-3.2.1 *Lovibond Tintometer*

C-3.2.2 *Glass Cell* — 5 ¼ inch size.

C-3.2.3 *Filter Paper*

C-3.3 Reagent

C-3.3.1 *Distilled Water*

C-3.4 Procedure

Prepare a solution of the product containing 5 percent active matter in distilled water. Filter the solution. Fill the 5 ¼ inch glass cell with the solution. Place the cell in position in the tintometer. Place along side of it red and yellow glass slides to match the colour of the sample, observing colour of the sample and of the combination of glass slides through the eye piece.

C-3.5 Report

Report the colour of the solution containing 5 percent active matter in terms of Lovibond units expressed as the sum of the yellow and five times red slides.

ANNEX D

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF TOTAL INORGANIC SALTS

D-1 REAGENTS

D-1.1 Ethyl Alcohol — *See* IS 321.

D-2 PROCEDURE

Weigh accurately about 10 g of the sample in a 250 ml beaker and digest with about 150 ml of ethyl alcohol on a steam bath. Filter into a filter flask through a dried and tared Gooch or sintered glass crucible (G-3 or G-4). Wash it several times with hot ethyl alcohol to remove all the alcohol solubles. Dry the crucible with residue at 105 ± 2° C for 3 h. Cool in a desiccator. Weigh the matter insoluble in alcohol.

D-3 CALCULATION

$$\text{Total inorganic salts, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of matter insoluble in alcohol, and
M_2 = mass in g of the material taken for the test.

ANNEX E

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF DISULPHONATE BY HIGH PRESSURE LIQUID CHROMATOGRAPHY

E-1 APPARATUS

E-1.1 High Pressure Liquid Chromatography System with Differential Refractometer Detector

E-1.2 Zorbax Column

E-1.3 Injector (with 20 µl Sample Loop)

E-2 REAGENTS

All solvents shall be of HPLC grade.

E-2.1 Mobile Phase — 0.4 M of sodium acetate in a mixture of 27.5 ml of methanol.

E-2.2 Acetonitrile — 37.5 ml.

E-2.3 Water — neutralized to *p*H 7.5 to 7.8 with glacial acetic acid, 35 ml.

E-3 PROCEDURE

E-3.1 Sample Preparation

Make 3 to 5 percent solution by mass on an 100 percent active basis in HPLC grade water. Filter through 0.45 micro millipore filter paper.

E-3.2 Vacuum filter mobile phase through 0.45 micro millipore filter paper.

Adjust attenuation and integrator for maximum peak presentation. Set the sample solution in 20 μl quantity.

E-3.3 Test Results

Since disulphonate is the most polar component in sodium alpha olefin sulphonate, it may eluate early in the reverse phase system just after the solvent peak. Distinct separation of disulphonate from other components is, therefore, possible. Express the peak area of the peak immediately after the solvent peak as disulphonate content.

ANNEX F

[*Table* 1, *Sl No.* (viii), (ix) *and* (x)]

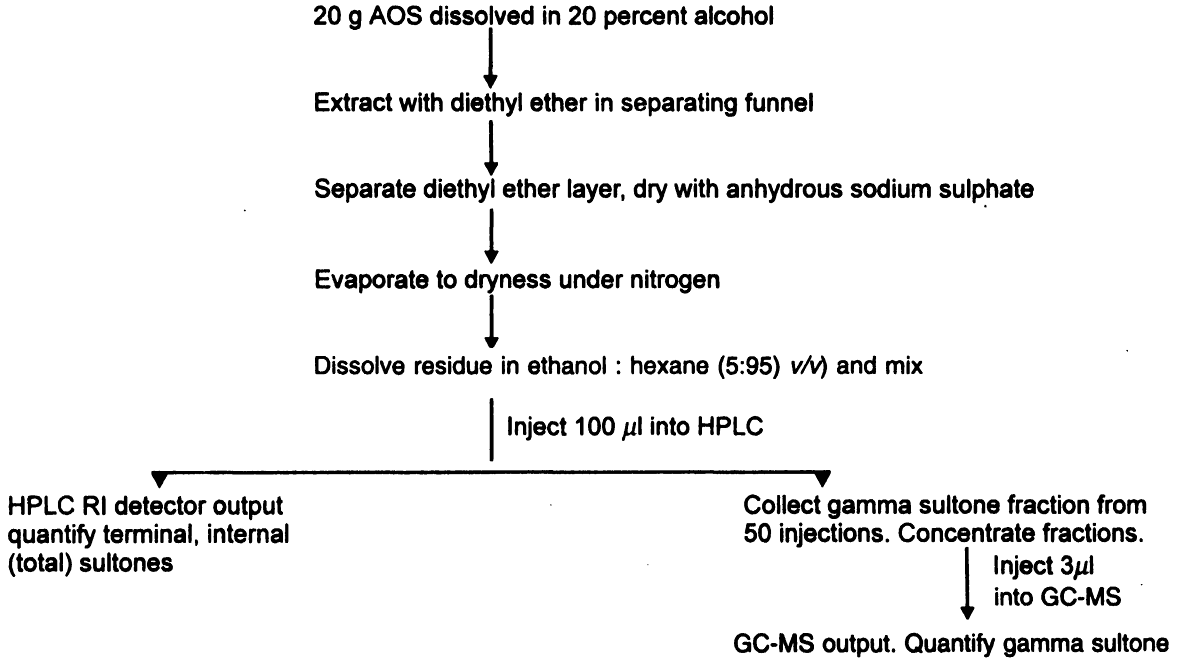
DETERMINATION OF SULTONES (TOTAL, INTERNAL AND GAMMA SULTONES)

F-1 OUTLINE OF THE METHOD

The method deals with the analysis of terminal and internal sultones, together termed as total sultones in AOS by High Performance Liquid Chromatography (HPLC) and 1-alkene 1-3 sultone (also known as gamma sultone) by Gas Chromatography-Mass Spectrometry (GC-MS). The HPLC method has a sensitivity to detect total and internal sultones in ppm levels while GC-MS can quantitatively determine 1-3 γ sultones in AOS at ppb levels.

In order to quantitatively estimate sultones in AOS, it is also necessary to use the appropriate standard sultones. Since these are not commercially available, detailed procedures for the laboratory synthesis and characterization of individual sultones are also described in Annex G.

F-2 ANALYTICAL STRATEGY


20 g AOS dissolved in 20 percent alcohol
Extract with diethyl ether in separating funnel
Separate diethyl ether layer, dry with anhydrous sodium sulphate
Evaporate to dryness under nitrogen
Dissolve residue in ethanol : hexane (5:95) v/v) and mix
Inject 100 µl into HPLC
HPLC RI detector output quantify terminal, internal (total) sultones
Collect gamma sultone fraction from 50 injections. Concentrate fractions.
Inject 3µl into GC-MS
GC-MS output. Quantify gamma sultone

F-3 APPARATUS

F-3.1 Separating Funnels — 250 ml capacity.

F-3.2 Beakers — 250 ml capacity.

F-3.3 Conical Flasks — 250 ml capacity.

F-3.4 Volumetric Flasks — 5 ml capacity.

F-3.5 Sample Filtration Kit (Millipore)

F-3.6 Rotavaporator

F-4 REAGENTS

F-4.1 Ethanol — 20 percent aqueous from absolute alcohol.

F-4.2 Sodium Sulphate — Anhydrous, analar grade.

F-4.3 Hexane — HPLC grade.

F-4.4 Diethyl Ether — HPLC grade.

F-4.5 Distilled Water — Glass distilled water.

F-4.6 Tetradecene — AR grade.

F-5 PROCEDURE

F-5.1 Calibration

Prepare 1 000 ppm stock solution of the standard sultones in hexane (HPLC grade). Take 1, 2, 4, 6 and 8 ml of the stock solution in 10 ml volumetric flasks and make up to the mark using hexane (HPLC grade). Inject 100 µl of each of these solutions containing 10, 20, 40, 60 and 80 µl respectively in the HPLC column and elute with the mobile phase. Draw calibration curves with area under the curve *vs.* amount of standard sultones in µg.

F-5.2 Apparatus and Operating Conditions

F-5.2.1 *HPLC with Data Processor*

F-5.2.2 *Refractive Index Detector (RID)*

F-5.2.3 *Column*

Either of the column mentioned below can be used:

Cyano propyl silyl (CPS) hypersil column	5 micron
Length	1 m
ID	4.6 mm

or

CPS—hypersil guard column	5 micron
Length	1 m
ID	4.6 mm

or

Finesil	5 micron
Length	1 m
ID	4.6 mm
Stationery phase	silica gel

F-5.2.4 *Mobile Phase*

Ethanol/*n*-hexane	5 : 95 (*v/v*)
Flow rate	1 ml/min
Detector sensitivity	16×10^{-6} RUIFS,
Sample loop	200 µl
Injector	100 µl loop

F-5.2.5 GC-MS — Any standard instrument equipped with single ion monitoring (SIM) and negative ion chemical ionization mode (NCI).

F-5.2.6 *Column*

DBFFAP column	
Length	30 m
ID	0.25 mm
Column temp.	220°C
Injection temp.	300°C
Separator temp.	300°C
Carrier gas	Helium
Flow rate	1 ml/min
Ionization mode	negative ion chemical ionization
Reagent gas	methane
Ionization potential	200 eV
Accelerating voltage	3 kV
Ion source temp.	180°C

F-5.3 Sample Preparation and Analysis

Dissolve AOS (20 g) in aqueous ethanol (100 ml, 20 percent (*v/v*) and extract with diethyl ether (3 × 30 ml). Combine the extracts and wash with distilled water (3 × 30 ml), dry over anhydrous sodium sulphate and filter through tightly packed cotton. Wash the inside of the separating funnel with 30 ml diethyl ether and filter through the same filter. Combine the extracts. Concentrate by evaporating the solvent by rotavaporation. Transfer the residue into a vial (5 ml) with a conical bottom and evaporate to dryness under nitrogen. Dissolve in ethanol/*n*-hexane (5/95, *v/v*) to make up to 5 ml. Filter the sample through the filtration kit (millipore) and then inject (100 µl × 50 injections) into the HPLC column. Elute with hexane containing 5 percent ethanol as the mobile phase and compare retention time of the eluted peaks with that of the standard sultones (*see* Annex G, Fig. 2 and 3).

For total and internal sultones, calculate the amount of particular sultone in the sample by measuring the area of the peak corresponding to that sultone and comparing that with calibration curve of the standard sultone. (*see* Annex G, Fig. 4).

For 1-alkene-1,3 sultones, collect the fraction having the same retention time as the standard in a flask for 3 min (starting at 1.5 min before the standard gamma sultone peak retention time). Concentrate the combined fractions in a 3 ml vial tube. Add 1-tetradecene (100 µl exactly) to this vial tube and the solution placed in an ultrasonic bath. This sample solution is now analyzed by GC-MS for quantitative determination of gamma sultone (*see* Annex G, Fig. 1).

Inject the sample solution 3 µl into GC-MS. In GC-MS, detect the molecular ions of each unsaturated sultone (ex C_{12} - 246; C_{14} - 274; C_{14} - 302) simultaneously, and record their peak ateas and retention times. Determine the contents of each unsaturated sultone from a calibration curve using standard unsaturated sultones (*see* Annex G, Fig. 5, 6, 7, 8 and 9).

F-5.4 Calculations

F-5.4.1 *For Total and Internal Sultones*

Calculate the sultone levels in the sample (in ppm) as follows:

$$\text{Sultone level on 100 percent active basis} = \frac{a \times V_1 \times 100\,000}{V_2 \times W \times D}$$

where

a = amount of sultone in µg obtained from calibration curve,

V_1 = total volume to which the extract is made up (ml),

V_2 = volume of the extract inject (µl),

W = mass of AOS used in g, and

D = a factor corresponding to percent active matter in AOS (to be ascertained by AD determination).

F-5.4.2 *For Gamma Sultone* (1 *-alkene*-1,3 *-sultones*)

$$C = [A \times 10^{-9} \times (100/V_3) \times 10^{9}]/(M \times D/100)$$

where

C = concentration of gamma sultone (alkene 1,3 sultone in ppb in active matter basis),

A = the amount (*n*g) of gamma sultone from calibration curve,
V_3 = injected volume (μl),
D = active matter content (percent), and
M = sample mass in g.

ANNEX G

(Supplement to Annex F)

LABORATORY SYNTHESIS AND CHARACTERIZATION OF INDIVIDUAL SULTONES

G-1 PROCEDURE FOR SYNTHESIS OF INTERNAL SULTONE

G-1.1 Apparatus

G-1.1.1 *Falling Film Reactor (FFR)—A* vertical glass tube 120 cm (4 ft) × 7 mm id surrounded by a cooling jacket.

G-1.1.2 *Separating Funnels*

G-1.1.3 *Beakers*

G-1.1.4 *Glass Column* — 60 cm (2 ft) approximately.

G-1.1.5 *Distillation Apparatus*

G-1.1.6 *Overhead Stirrer*

G-1.1.7 *3-Necked Round Bottom Flask*

G-1.1.8 *Vigreux* — 80 cm (3 ft) approximately.

G-1.1.9 *Preparative HPLC* (for example, Water Prep 500 HPLC)

G-1.2 Reagents

G-1.2.1 *Oleyl Alcohol* — Reagent grade.

G-1.2.2 *p-Toluene Sulphonyl Chloride* — Reagent grade.

G-1.2.3 *Sodium Hydroxide* — Reagent grade.

G-1.2.4 *Tetra Butyl Ammonium Iodide* — Reagent grade.

G-1.2.5 *Hexane* — Reagent grade.

G-1.2.6 *Hexane* — HPLC grade.

G-1.2.7 *Anhydrous Magnesium Sulphate* — Reagent grade.

G-1.2.8 *Dry Tetrahydrojuran* — Reagent grade.

G-1.2.9 *Lithium Aluminium Hydride* — Reagent grade.

G-1.2.10 *Dry Sulphur Trioxide*

G-1.2.11 *Dry Nitrogen*

G-1.2.12 *Silica for Column* — 100-200 mesh.

G-1.2.13 *Diethyl Ether* — Reagent grade.

G-1.2.14 *Diethyl Ether* — HPLC grade.

G-1.2.15 *Ethanol* — 50 percent aqueous.

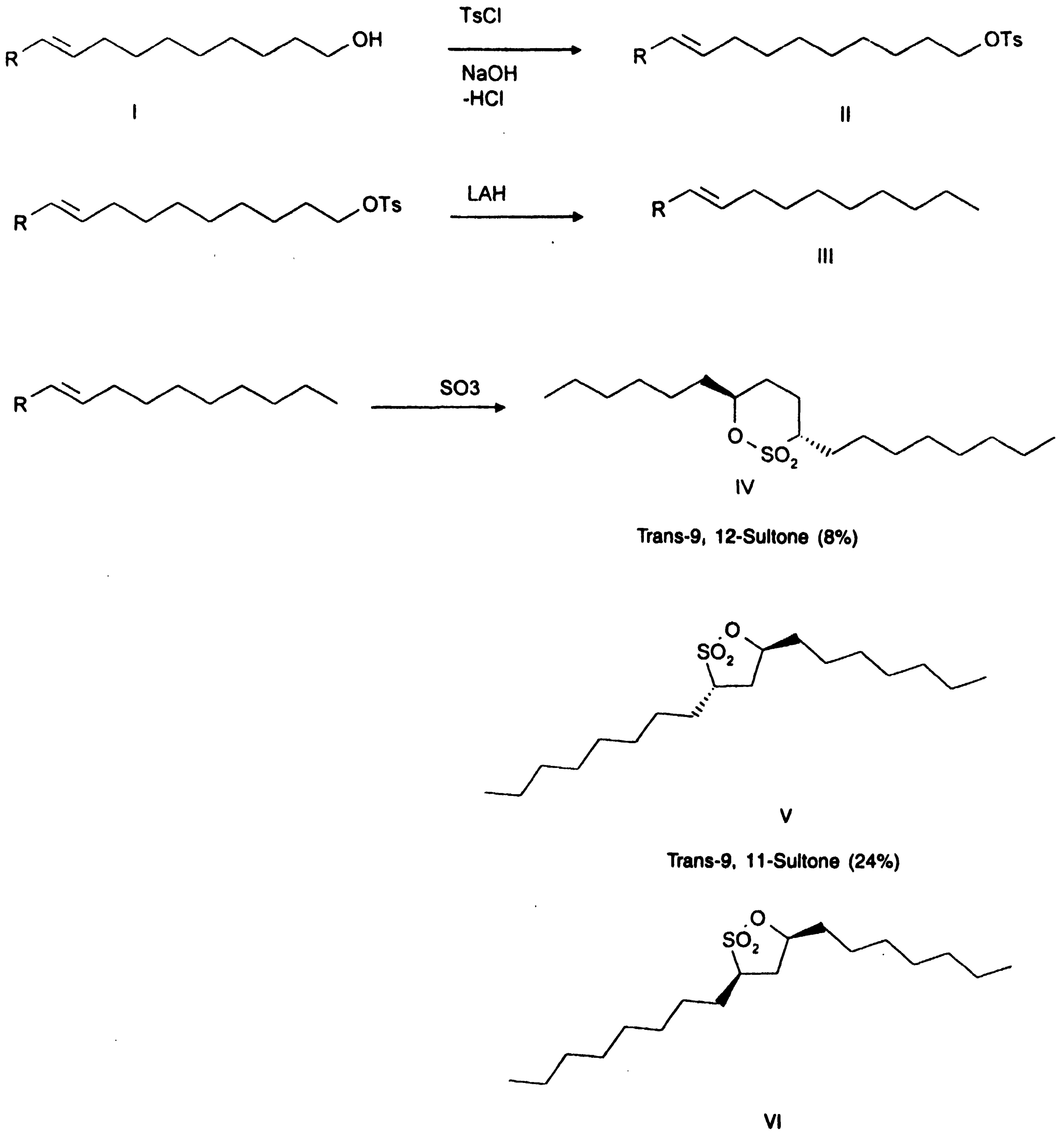
G-1.2.16 *Distilled Water*

G-1.3 Procedure

Treat oleyl alcohol (I) with 12 percent molar excess of *p*-toluene sulphonyl chloride in the presence of water (300 ml), sodium hydroxide (4 moles) and tetra butyl ammonium iodide (0.02 mole). Stir the mixture vigorously for 4-5 min whereupon an exothermic reaction takes place raising the temperature of the mixture to 90°C. Stir the material for a further one-and-a-half hours and extract the crude tosylate (II) with hexane after the addition of water (600 ml) and dry over anhydrous magnesium sulphate. Dissolve yield of crude tosylate – 770 g molar excess of lithium aluminium hydride in THF. After addition of water and base, remove the insoluble lithium and aluminium salts and discard. Remove the filtrate with water and evaporate to give the crude 9-octadecene (III) (80 percent pure by GC). Purify the crude olefin by distillation using 3 ft vigreux (133°C/0.5 mm). Sulphonate (III) in the falling film reactor. Supply the olefin to the top of the tube and drive downward by a stream of nitrogen. Introduce a mixture of gaseous sulphur trioxide and dry nitrogen (4:96) near the top of the tube. The mole ratio of sulphur trioxide to olefin (1.22:1) and the cooling jacket temperature at 35°C. The throughput of olefin should reach 5.8 g/min and film residence time was 60 seconds.

Leave the sulphonation product (140 g) for 21 days at 20-25°C, then neutralize with 5 percent aqueous sodium hydroxide and extract with hexane. Wash the hexane extract with 50 percent aqueous ethanol to remove residual sulphonate salts and elute through a 2 ft silica column (100-200 mesh) with 20 percent diethyl ether in hexane. Separate the eluate (30 g) by preparative HPLC using Water Prep 500 instrument (stationary phase: silica, mobile phase; hexane: diethyl ether 95:5). The sultones isolated in this way are *trans* 9, 12 sultones (IV, 2.5 g, 8 percent), *trans* 9, 11 sultone (V, 7.5 g, 24 percent) *cis* 9, 11 sultone (VI, 2.5 g, 8 percent). recrystallized from methanol and identified by $^{1)}$H NMR spectra (supported by evidence in MS and IR spectroscopy).

G-1.4 Scheme for Synthesis



Trans-9, 12-Sultone (8%)

Trans-9, 11-Sultone (24%)

Cis-9, 11 Sultone (18%)

G-1.5 Identification of IV, V and VI

G-1.5.1 *Mass Spectrometry*

A molecular ion at m/e 332 corresponding to a molecular formula of $C_{18}H_{36}SO_3$ was observed for each compound as expected for C_{18} internal sultones. It may also be found that V and VI show very similar fragmentation patterns.

G-1.5.2 *^{1}H NMR and Infrared Spectrometry*

G-1.5.2.1 *Structure IV*

The proton spectrum of IV has two broad absorptions at 4.5 δ Ha, and 2.8. δ, Hb, corresponding to 1 proton each. Further absorptions at 2.1, 1.9 and 1.8 are tentatively assigned to Hc. Hd and Hc although integrations are not obtainable due to the large alkyl chain absorptions at 1.25 δ.

The typical δ sultone absorption at 915 cm^{-1} as found for 1,4-sultone could be observed but also further absorptions at 880 and 820 cm^{-1} S=O symmetrical and asymmetrical stretches can also be seen at 1 360 and 1 175 cm^{-1}, the latter being a very sharp absorption.

G-1.5.2.2 *Structures V and VI*

a) The ^{1}H NMR spectra of V and VI are substantially different as shown below:

Proton	*Shift*	*(b)Integration*	*Peak Type*
V Ha	4.6	1	"Quintet"/Doublet of triplets
V Hb	3.3	1	"Quintet"/Doublet of triplets
V Hc	2.4	2	AB quartet (JAB~7.5 Hz)
VI Ha	4.5	1	Multiplet
VI Hb	3.3	1	Multiplet similar to Ha
VI Hc	2.6	1	Multiplet
VI Hd	2.0	1	Multiplet. possible doublet of doublets

VI and V are assigned as *cis* and *trans* isomers of 9,11-octadecane sultone as shown in **G-1.4.**

b) The infra-red spectra of V and VI are almost identical. Both sultones have strong absorptions at 1 340 and 1 170 cm^{-1}, the S=O symmetrical and asymmetrical stretches, and medium absorptions at 975 and 830 cm^{-1} typical of *γ*sultones. There are, however, a noticeable difference in the relative intensities of the latter two isomers. In V, the 830 cm^{1} band is much more intense than the 975 cm^{-1} band whereas in VI, the 2 bands are of roughly equal intensity.

Protons Hc in V are in very similar chemical environments thus the resonance for these protons appear as a doublet of doublets at Hb, whereas Hc and Hb in VI can be seen to be *cis* and *trans* respectively to the alkyl chains. This results in the protons appearing approximately equi-distant, either side of Hc absorption observed for Hc in V. The changes in complexity of the absorptions of Ha and Hb in V and VI are also in accord with the structural assignments. The ^{13}C NMR spectra are also consistent with the proposed structures.

Melting points of IV, V and VI

N	52.5°C ± 0.2°C
V	32.2°C ± 0.2°C
VI	42.1°C ± 0.2°C

G-2 PROCEDURE FOR SYNTHESIS OF TERMINAL 1,4 SULTONES

G-2.1 Apparatus

G-2.1.1 *Falling Film Reactor (FFR)* — Vertical glass tube 120 cm (4 ft) × 7 mm id surrounded by a cooling jacket.

G-2.1.2 *Separating Funnels*

G-2.1.3 *Conical Flasks*

G-2.1.4 *Beakers*

G-2.1.5 *Glass Column 60 cm* (*2 ft*)

G-2.2 Reagents

G-2.2.1 *1-Hexadecene* — Reagent grade.

G-2.2.2 *Dry Sulphur Trioxide*

G-2.2.3 *Dry Nitrogen*

G-2.2.4 *Sodium Hydroxide* — 10 percent aqueous (*v/v*).

G-2.2.5 *Ethanol* — 50 percent aqueous.

G-2.2.6 *Petroleum Ether* — (40-60°C) Reagent grade.

G-2.2.7 *Hexane* — Reagent grade.

G-2.2.8 *Hexane* — HPLC grade.

G-2.2.9 *Diethyl Ether* — HPLC grade.

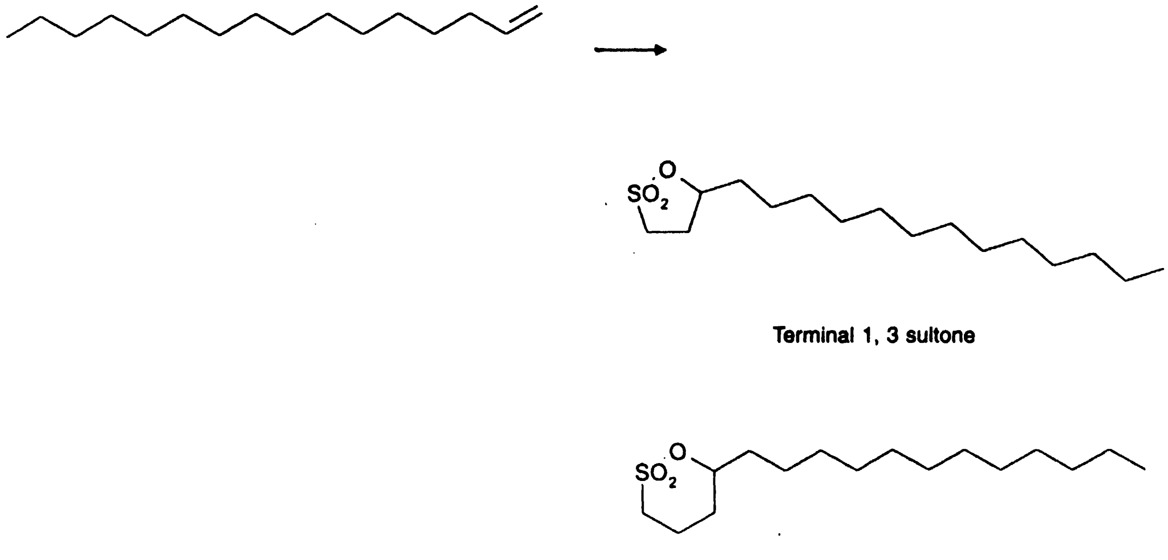
G-2.2.10 *Diethyl Ether* — Reagent grade.

G-2.2.11 *Distilled Water*

G-23 Procedure

Sulphonate the 1-Hexadecene in the FFR. Supply the olefin to the top of the tube and drive downwards by a stream of dry nitrogen whilst gaseous sulphur trioxide/nitrogen (4:96) introduce near the top of the tube. Maintain the mole ratio of sulphur trioxide to olefin (1.20:1), the cooling jacket temperature at 45°C, the throughput of olefin ~ 5 g/min and the film residence time at 60 seconds.

Collect the product emerging from the reactor in over a period of 10 min into aqueous 10 percent sodium hydroxide. Mix sample (50 g) of the aqueous neutralizate with ethanol (100 ml). Wash the resulting solution with 50 percent ethanol (2 × 50 ml) and evaporate. The residue was loaded onto a 2 ft silica column (100 - 200 mesh), elute with 20 percent diethyl ether in hexane.



Terminal 1, 3 sultone

Terminal 1, 4 sultone

Separate the eluate by preparative HPLC using a preparative HPLC instrument (Stationary phase: Silica, mobile phase hexane: Diethyl ether 95 : 5) and identify by 'HNMR spectra (supported by evidence in mass spectrometry and infra-red spectroscopy).

G-2.4 Scheme for Synthesis

G-2.5 Identification of Terminal -1,4 Sultones

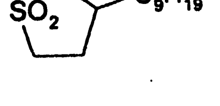
G-2.5.1 *'HNMR*

It has a broad absorption at 4.6 δ-Ha, 2.9 and 3.2 δ due to Hd and Hc, 2.2 δ-Hc and 1.6 δ due to Hc. The alkyl chain absorption is at 1.25 δ. This confirms the structures above.

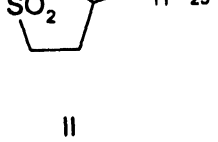
G-3 PROCEDURE FOR THE SYNTHESIS OF — ENE (GAMMA) SULTONES

2-Dodecene-1 -O1 is treated with PBr_3 in pyridine, and the products converted by the action of Na_2SO_3 into sodium 2-dodecene-1-sulphonate. Reaction of the latter with aqueous bromine afforded 2-bromo-l,3-dodecanesultone. For dehydrobromination, a mixture of 2.75 g of the bromo-sultone and 2.91 g freshly distilled collidine is heated at 100°C under nitrogen for one hour; white solid is gradually formed in the initially clear liquid. After cooling, the material is dissolved in 600 ml 1 : 1 hexane:ether, and washed 5 times with 400 ml 0.01 N HCl and three times with distilled water. After drying over Na_2SO_4, the solvents are evaporated to leave 2.11 g (98 percent) of white crystals. A portion of the product, l-dodecene-l,3-sultone, was purified by TLC (M.P.37.0-37.5°C; S : 13.00,12.89 calculated for $C_{12}H_{22}O_3S$:C; 58-50; H: 9.00, O : 19.48; S: 13.02). The NMR, mass, and infra-red spectra of these products are shown in the attached figures. They are consistent with the assigned structure shown below. The NMR spectrum of the synthetic l-dodecene-l,3-sultone showed peaks identical to those attributed to an unsaturated *y*-sultone. It is concluded that the structures from NMR, IR and mass spectrometry, synthesized were l-dodecene-l,3-sultone (I) and 1-tetradecene-l,3-sultone (II).

Structures



I



II

REPRESENTATIVE HPLC & GC CHROMATOGRAMS & STANDARD CALIBRATION PLOT FOR SULTONES

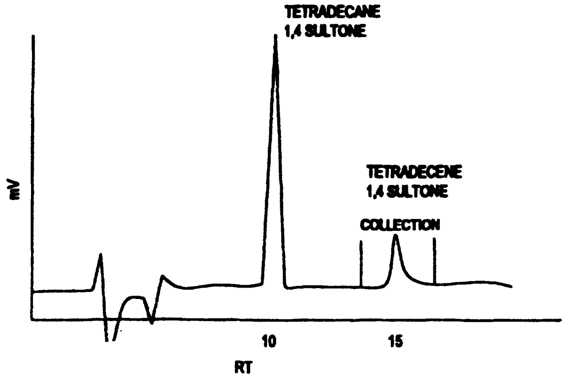


Fig. 1 1 Alkene - 1, 5 Sultone

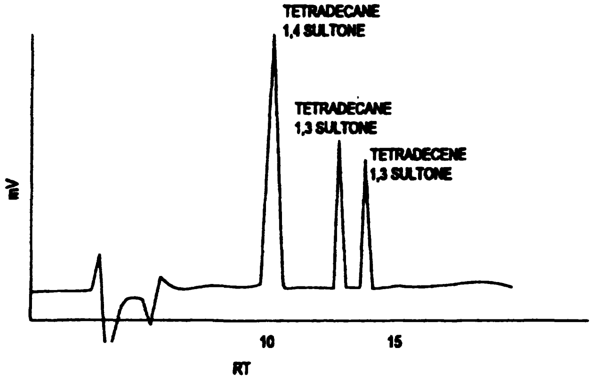


Fig. 2 1 Alkene - 1, 3 Sultone

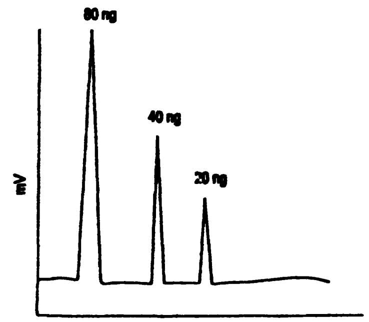


Fig. 3 1 Alkene - 1, 3 Sultone

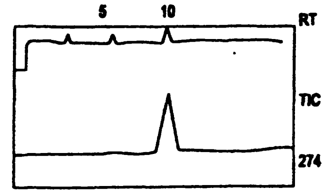


Fig. 7 Usaturated Sultone

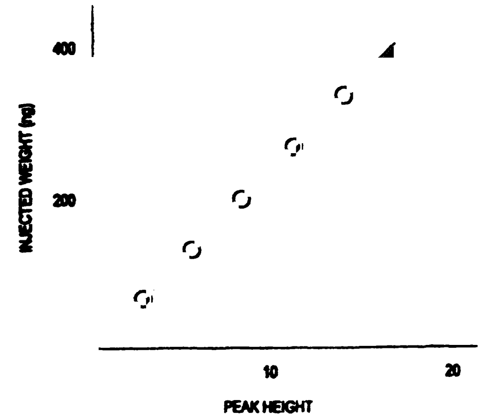


Fig. 4 Caliberation Curve of Standard Sultone (For Total and Internal Sultone)

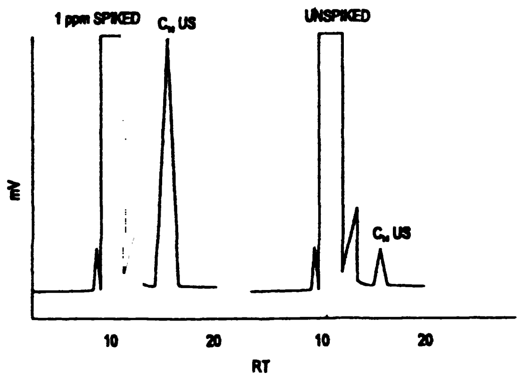


Fig. 5 Unsaturated Sultone

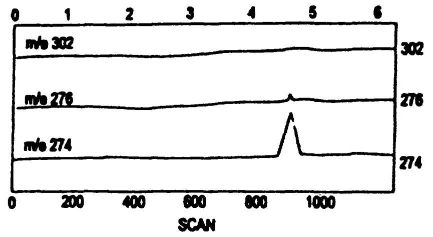


Fig. 9 Unsaturated Sultone

GCMS

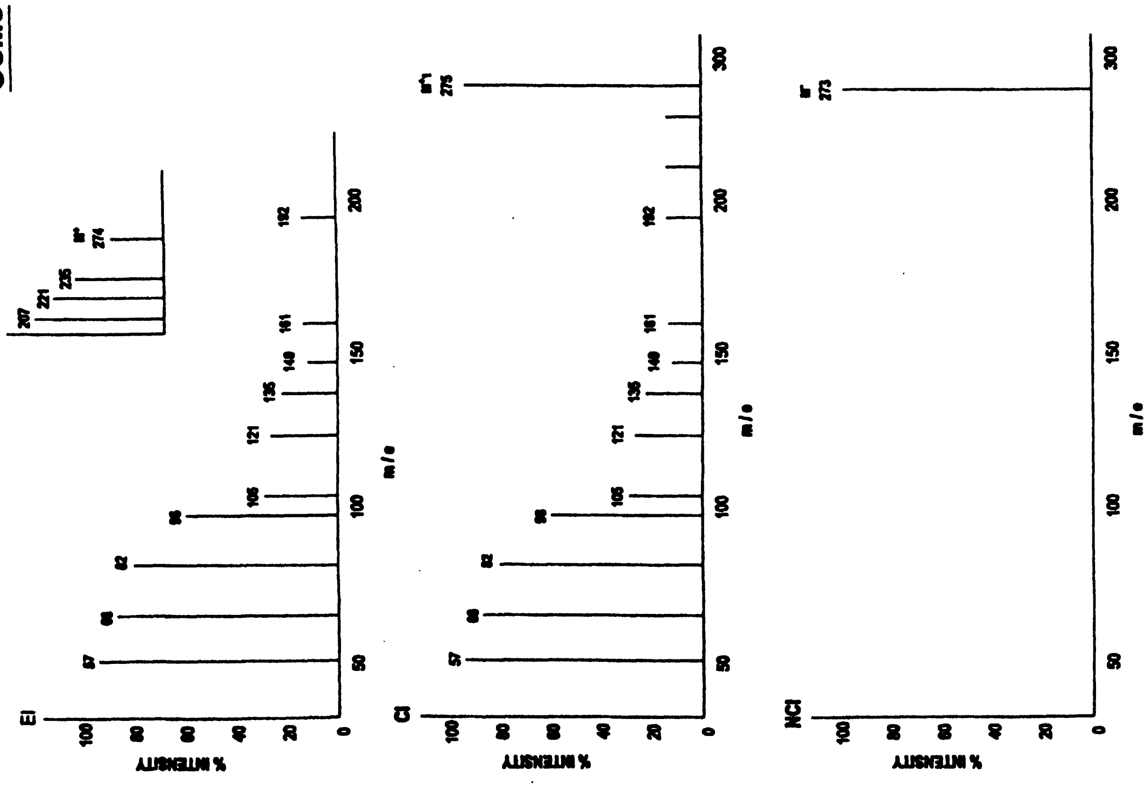


FIG. 6 UNSATURATED SULTONE

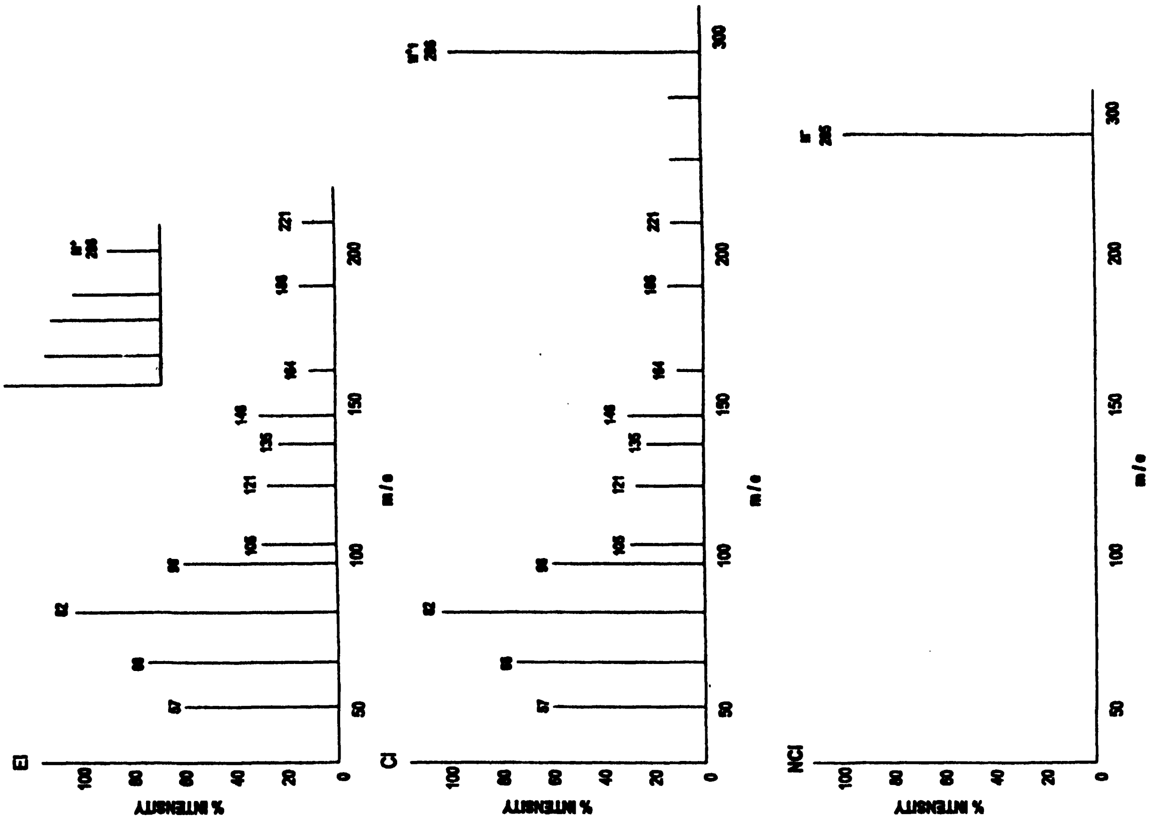


FIG. 8 UNSATURATED SULTONE

ANNEX H

(Foreword)

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(*Continued on page* 14)

(*Continued from page* 13)

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